DOCKET NO: 204628US0

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

MICHIYASU KOMATSU : EXAMINER:

SERIAL NO: 09/805,035 :

FILED: MARCH 14, 2001 : GROUP ART UNIT: 1755

FOR: SILICON NITRIDE WEAR RESISTANT MEMBER AND MANUFACTURING METHOD THEREOF :

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

I, Mr. Michiyasu Komatsu, declare and state as follows:

1. I am the named inventor of the above-identified application.

2 I am familiar with the claims, and have read the Office Action mailed December 16, 2003 in the above-identified application.

3. The present invention is characterized in that a ball-shaped wear resistant member has a rolling fatigue life of 400 hr or more. Such an excellent rolling fatigue life is based on the titanium nitride particles having a long axis in the range from 0.04 to 1 μm and an aspect ratio in the range from 1.0 to 1.2 (at least 80% by volume of particles), and β-Si_3N_4 phase as a main phase of the silicon nitride sintered body. The rolling fatigue life of the ball-shaped wear resistant member is influenced by the form of the titanium nitride particles and the main phase of the silicon nitride sintered body (β-Si_3N_4 phase or α-Si_3N_4 phase).

4. Tables 1 and 2 attached herewith represent experiments conducted under my supervision and/or control. The Tables show the relationship between rolling fatigue life and the main phase of the silicon nitride sintered body. Table 2 shows that silicon nitride sintered bodies having α-Si_3N_4 phase are inferior with respect to rolling fatigue life.

5. The following discussion of Embodiments is in connection with the Embodiments described in the specification of the above-identified application.

6. Experimental data (Table 1)

The form of Embodiment 2 (ball) is different from the form of Embodiment 1 (circular board), but Embodiment 2 is made under conditions (same composition, same degreasing condition, same heat-treatment condition, same sintering condition and same HIP condition) identical to Embodiment 1. Therefore, the main phase of the Si_3N_4 sintered body in Embodiment 1 consists essentially of β-Si_3N_4 phase in the same way as Embodiment 2.

In Table 1, under the heading "Embodiment," the first number relates to a "ball" embodiment, while the number in parenthesis relates to a "circular board" embodiment, but which is otherwise identical, as detailed above.

Part of the data in Table 1 can be found in Tables 3 and 4 of the specification.

Experimental data (Table 2)

The data in Table 2 was carried out as follows:

First, the raw materials having the composition shown in the Table 2 were made. The raw materials contain organic binder. The raw materials, after being preliminarily molded spherical by means of a mold, underwent cold isostatic pressing under a pressure of 98 MPa to prepare spherically molded bodies of a diameter 11 mm.

These molded bodies were degreased at 450°C in a stream of air for 4 hr, thereafter followed by sintering in a nitrogen gas atmosphere under the conditions shown in the Table.

The obtained sintered bodies were HIP treated under the conditions shown in the Table. The sintered bodies after the HIP treatment were polished into balls of a diameter 9.52 mm and a surface roughness Ra of 0.01 μm to prepare samples.

In the samples (ball-shaped Si_3N_4 sintered bodies), the respective amounts of α-Si_3N_4 phase and β-Si_3N_4 phase in the Si_3N_4 sintered bodies were measured by x-ray diffraction. Furthermore, crushing strength at room temperature, fracture toughness due to a microindentation method, and rolling fatigue life were measured in the same way as described for Embodiment 2 in the specification. These measurements are shown in Table 2.

7. The samples shown in Table 2 have a α-Si_3N_4 phase. Table 2 shows characteristics of the silicon nitride sintered bodies having a α-Si_3N_4 phase. The rolling fatigue life for all the silicon nitride sintered bodies shown therein is shorter than 400 hours due to the silicon nitride sintered bodies having a α-Si_3N_4 phase.

8. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

9. Further declarant saith not

Michiyasu Komatsu
Signature

2004 March, 11
Date

Received at: 4:08AM, 3/11/2004

2004-03-11 18:10 宛先-OBLON 殿 送信元-ｻｸﾗ国際特許事務所 T-064 P.006/007 U-950

Table 1

Embodiment	Raw Material Composition (% by mass)								Sintering Conditions		HIP Conditions			Constituting Phase (%)		Crushing Strength (MPa)	Fracture Toughness ($MPa \cdot m^{1/2}$)	Rolling Fatigue Life (hr)
	Si_3N_4	Rare Earth Oxide		Al_2O_3	AlN	TiN Source		MgO	Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)	Pressure (atm)	β-Si_3N_4	α-Si_3N_4			
2 (1)	87.5	Y_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	270	7.3	>400
26 (3)	87.5	Y_2O_3	5	3	3	TiO_2	1.5	–	1750	4	–	–	–	100*	–	245	6.9	>400
27 (4)	88.8	Y_2O_3	5	3	3	TiO_2	0.2	–	1750	4	1700	1	1000	100*	–	260	6.7	>400
28 (5)	84.0	Y_2O_3	5	5	3	TiO_2	5	–	1750	4	1700	1	1000	100*	–	285	7.1	>400
29 (6)	86.9	Y_2O_3	5	2	5	TiO_2	3	–	1850	4	1800	1	1000	100*	–	230	6.8	>400
30 (7)	86.5	Y_2O_3	5	2	2	TiO_2	1.5	–	1750	4	1700	1	300	100*	–	265	6.9	>400
31 (8)	86.5	Y_2O_3	5	5	2	TiO_2	1.5	–	1600	4	1600	1	1000	100*	–	275	6.7	>400
32 (9)	86.5	Y_2O_3	5	–	5	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	300	7.2	>400
33 (10)	89.5	Y_2O_3	5	–	–	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	280	6.8	>400
34 (11)	97.0	Y_2O_3	0.5	5	–	TiO_2	0.5	–	1900	4	1850	1	1000	100*	–	240	6.7	>400
35 (12)	75.0	Y_2O_3	10	3	5	TiO_2	5	1	1700	4	1600	1	1000	100*	–	235	6.6	>400
36 (13)	87.0	Y_2O_3	5	3	3	TiO_2	1	2	1700	4	1600	1	1000	100*	–	285	7.1	>400
37 (14)	86.0	Y_2O_3	5	3	3	TiO_2	1	2	1750	4	-	-	-	100*	–	250	6.7	>400
38 (15)	86.0	Y_2O_3	5	3	3	TiO_2	1	–	1700	4	1650	1	1000	100*	–	289	6.9	>400
39 (16)	87.5	Y_2O_3	5	3	3	TiC	1.5	–	1750	4	1700	1	1000	100*	–	270	7.1	>400
40 (17)	87.5	Y_2O_3	5	3	3	TiN	1.5	–	1750	4	1700	1	1000	100*	–	255	6.8	>400
41 (18)	87.5	Y_2O_3	5	3	3	TiB_2	1.5	–	1750	4	1700	1	1000	100*	–	260	6.6	>400
42 (19)	87.5	Y_2O_3	5	3	3	$TiSi_2$	1.5	–	1750	4	1700	1	1000	100*	–	260	6.5	>400
43 (20)	87.5	Ce_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	275	6.7	>400
44 (21)	87.5	Nd_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	270	6.7	>400
45 (22)	87.5	Sm_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	255	6.6	>400
46 (23)	87.5	Dy_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	285	6.9	>400
47 (24)	85.0	Er_2O_3	7.5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	290	7.0	>400
48 (25)	87.5	Yb_2O_3	5	3	3	TiO_2	1.5	–	1750	4	1700	1	1000	100*	–	270	6.7	>400

*: Si_3N_4 grains consist essentially of β-Si_3N_4.

Received at: 4:08AM, 3/11/2004
2004-03-11 18:11 宛先-OBLON 殿 送信元-サクラ国際特許事務所 T-064 P.007/007 U-850

Table 2

Sample	Raw Material Composition (% by mass)						Sintering Conditions		HIP Condition			Constituting Phase (%)		Crushing Strength (MPa)	Fracture Toughness ($MPa\cdot m^{1/2}$)	Rolling Fatigue Life (hr)
	Si_3N_4	Y_2O_3	Al_2O_3	AlN	TiO_2	MgO	Temperature (°C)	Time (hr)	Temperature (°C)	Time (hr)	Pressure (atm)	β-Si_3N_4	α-Si_3N_4			
1	88.5	5	2	2	1.5	1	1500	4	1500	1	1000	78	22	200	5.8	275
2	88.5	5	2	2	1.5	1	1550	4	1500	1	1000	85	15	240	6.1	300
3	88.5	5	2	2	1.5	1	1600	4	1600	1	1000	97	3	260	6.5	375
4	89.5	5	3	--	1.5	1	1500	4	1600	1	1000	90	10	260	6.3	325
5	89.5	5	3	-	1.5	1	1500	4	1600	1	1000	97	3	265	6.5	375
6	89.5	5	2	2	1.5	0	1500	4	1500	1	1000	50	50	160	5.0	75
7	89.5	5	2	2	1.5	0	1550	4	1500	1	1000	74	26	235	6.2	250
8	89.5	5	2	2	1.5	0	1600	4	1600	1	1000	95	5	260	6.5	350
9	86.5	5	5	2	1.5	0	1500	4	1500	1	1000	65	35	175	5.6	150